18 X-RAY POWDER DIFFRACTOMETER (XRD)

A. X-ray film badges are worn by all operators and used to monitor the laboratory area near the instrument to detect leakage and exposure.

B. All maintenance and calibration data is recorded in a log and located near the equipment.

C. Day of Use QC:

a. A silicon dioxide (SiO_2) standard is run day of use prior to case work. The intensity in counts per second and the *d*-spacings are recorded at generator settings of 45*kV* and 40*mA* for the 26.64 peak in SiO_2. The value obtained at this angle must fall within +/- 0.03 of the stated value for the instrument to be used in case work. A value outside this range will necessitate a call to a service technician. The intensity in counts per second is compared to the values previously recorded for each recent day of use. If the operator notes a sudden decrease in intensity or questions these comparative values, the alumina standard should be run.

b. Resolution is checked by running a silicon dioxide (SiO_2) standard at generator settings of 45*kV* and 40*mA*. Enhancement of the 67^o - 69^o range of the SiO_2 pattern should reveal five distinguishable peaks. If five distinguishable peaks are obtained, then a check mark is placed in the appropriate column on the log sheet. A service technician will be called if these five peaks are not distinguishable.

c. The standard method showing the instrument conditions and settings is included with this Appendix. An XRD conditions sheet, with any modifications of the standard method noted, will be included with each case analyzed.

D. Monthly QC:

a. An alumina standard (NIST reference standard) is run monthly (only necessary on months XRD is needed for casework). The intensity in counts per second and the d-spacings are recorded at generator settings of 45*kV* and 40*mA* for the 43.39 and 76.90 peaks. The values obtained at these two angles must fall within ±0.03 of these stated values for the instrument to be used in casework. Values outside this range will necessitate a call to a service technician. When intensity values for the 43.39 peak and the 76.90 peak fall below 1400 counts per second and 850 counts per second respectively, the x-ray tube must be replaced.

♦End